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by

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13. ABSTRACT

Intercomparisons between flow stress, microhardness, and rosette size measurements for purified KCl single crystals, KCl:Ca, KCl:Sr, and KCl_xBr_{1-x} mixed single crystals are made. A good correlation between the flow stress and inverse of the length of the dislocation rosette size was found for this crystal system. Our results, along with the results of other investigators (1,2,3) show that once a rosette size versus flow stress calibration has been made for a particular crystal system, the rosette size becomes a useful, simple, nearly nondestructive technique for determining the flow stress of a crystal.

14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Alkali Halides						
Pure and doped KCl						
KCl:KBr mixed crystals						
Flow Stress						
Vickers Hardness						
Wing Size						

INTRODUCTION

This project was initiated to study mechanical hardening effects of impurities and ionizing radiation on alkali-halide laser-window materials. This report describes work done on the mechanical strength of KCl:pure, KCl:Ca, KCl:Sr and KCl_xBr_{1-x} single crystals for the period ending 31 October 1972.

Microhardness tests and flow stress measurements are techniques commonly used to determine the mechanical strength of a material. Davisson and Vaughan (1) have shown that the size of the dislocation rosette produced around a microhardness indentation is a useful and convenient test for determining mechanical strengths of single crystals since the size of the rosette should be inversely proportional to the flow stress and since this test can be performed at essentially the same time as the microhardness measurements. Groves and Fine (2) and Davidge (3) have used the rosette size to measure the strength of Fe doped MgO. Davidge found that for heavily doped material the rosette size was indeed inversely proportional to the flow stress in KCl. Although hardening mechanisms have been studied using the rosette size (4) few intercomparisons between flow stress, microhardness, and rosette size have been made on the same samples. KCl is an excellent material for an investigation of techniques for measuring mechanical strengths, since large pure or doped single crystals are readily available and since considerable work on the mechanical properties of pure and doped KCl has already been done (5,6,7). The purpose of this paper is to present flow stress, Vickers microhardness, and dislocation rosette size measurements for purified KCl single crystals, KCl:Ca, KCl:Sr, and KCl_xBr_{1-x} mixed single crystals.

EXPERIMENTAL PROCEDURE

The purified KCl and the Ca and Sr doped KCl samples were obtained from Czochralski grown crystal boules which were eight years old (8). Therefore, the results from these samples may not be typical of those for freshly grown crystals since some precipitation of the dopants may have occurred. The dopant levels listed in Table I are for the crystal boules and may differ somewhat from the dopant levels in the tested samples. The dopant level, however, should be nearly uniform for the three different mechanical strength tests since these tests were run on the same sample.

The flow stress was measured by compressing the samples along a <100> direction using an Instron testing machine at a crosshead speed of 0.5 cm/min. This corresponds to a strain rate of approximately 10^{-3} s^{-1}. To obtain the flow stress values shown in Table I, at least five freshly cleaved samples, each measuring approximately 2 mm x 2 mm x 10 mm, were tested and the measured flow stress values averaged. The resolved flow stress, τ_r, represents the lowest stress necessary to move fresh dislocations along the <110> primary slip direction (9). For alkali halide crystals, τ_r is $\frac{1}{2}\tau_o$, where τ_o was taken to be the stress value at the intersection of the tangents drawn to the elastic and first plastic portions of the stress versus strain curve. The values of τ_r in Table I are in good agreement with those obtained earlier by Sibley (10), who measured τ_r for samples of Ca and Sr doped KCl from the same boules used for this work.

A Leitz Miniload tester with a Vickers type diamond indenter was used for the microhardness and rosette (wing) size tests. In order to facilitate dislocation etching the indentations were made on freshly cleaved {100} faces in a glove box in which the absolute humidity was held under 8 g/m^3. The diagonals of the indentations were along the <100> directions. The

TABLE I

RESOLVED FLOW STRESS, τ_r; VICKERS HARDNESS, HV; AND WING SIZE, W_o

Material	Dopant (ppm)	τ_r (kg/mm^2)	τ_r (psi)	HV (kg/mm^2)	W_o (μm)
KCl-P-1	Purified	0.11	160	9.2	91
KCl-P-2	Purified	0.15	220	9.0	91
KCl-Ca-1	100 Ca	0.20	290	11.2	77
KCl-Ca-2	540 Ca	0.55	800	16.7	41
KCl-Sr-1	50-150 Sr	0.23	330	13.0	77
KCl-Sr-2	470 Sr	0.57	830	16.9	41
KCl-Sr-3	138 Sr	0.38	540	14.9	60
KCl 33% - KBr 67%		0.89	1300	21.2	24
KCl 75% - KBr 25%		0.96	1400	19.8	24

hardness numbers (Table I) were obtained from an average of at least five indentations for loads of 5 g, 10 g, 15 g, and 25 g; these hardness numbers were found to be essentially independent of load for loads of 5 g to 50 g.

The dislocation rosettes (Fig. 1) around the indentations were revealed by etching the sample momentarily (1 s to 3 s), rinsing it in acetone, and then blotting it dry. Glacial acetic acid was found to be the best etchant for the pure materials while a saturated solution of $BaBr_2$ in absolute methanol was more effective for the doped samples. The rosette size, or wing length, W, was taken as the length from the center of the indentation to the wing tip along a <110> direction.

RESULTS AND DISCUSSION

The relationship between the Vickers hardness number, HV, and the resolved flow stress, τ_r, measured on the same sample of each of the different crystals including a pure KBr crystal is portrayed in Figure 2. It is not surprising that HV is not directly proportional to τ_r since the indenter activates both the primary <110> and secondary <100> slip systems while the resolved flow stress measures only the stress necessary to move dislocations along the primary slip system. The hardness numbers and flow stress values for the purified and doped KCl are in substantial agreement with those reported by Chin et al. (11).

Figure 3 indicates that the dislocation rosette size, W, is directly proportional to the load on the indenter. Inspection of the figure suggests that there should be a finite wing size, W_o, for zero indenter load. This result can be qualitatively understood when the nature of the microhardness test is considered. In this test, a dead-weight loaded pyramidal diamond indenter is allowed to slowly contact the surface of the crystal. The indenting diamond generates a string of fresh dislocations. The first of these dislocations is forced out by succeeding ones to a distance for which the applied stress on the string just equals the minimum stress needed to move dislocations along the primary slip direction, τ_r. Thus, the greater τ_r, the smaller W. The maximum stress on the crystal occurs at the instant the indenter contacts the sample and the dislocations immediately move out a distance W_o, since the dislocation velocity is very much greater than the indenter velocity (5). This conjecture is further substantiated by the results of E. Aerts et al. (12) who, while studying the mobility of dislocations in X-irradiated NaCl, established that the wing size is independent of the orientation of the

indentation figure. Any additional wing extent, $W-W_o$, comes about only because the indenter displaces crystal material as it moves on into the crystal. The indenter stops penetrating the crystal when the indenter stress is balanced by the rupture stress of the crystal; hence, the initial rosette size, W_o, is the one to be compared to the resolved flow stress. Figure 4 is a plot of the resolved flow stress, τ_r, versus $100/W_o$. It clearly shows τ_r to be inversely proportional to W_o for larger τ_r values. The data seem to indicate that the wing sizes approach some maximum value for the softer crystals. Since the wing lengths for the softer crystals are greater than 100 μm, subgrain boundaries or other macroscopic crystal defects could easily be limiting the extent of the wings. Davidge's results on MgO:Fe also indicated that the flow stress is inversely proportional to the wing size except at low flow stresses where the wing size is smaller than would be expected.

For a given indenter load, τ_r is inversely proportional to W for the harder crystals, but the linear region of τ_r versus W^{-1} does not intercept the origin. This fact, combined with the fact that deviations occur for large W (soft crystals) indicates that care should be exercised when using only the dislocation rosette size technique to investigate hardening mechanisms.

SUMMARY

For certain applications it would be useful to have a nondestructive test for the mechanical strength of a material. We have shown that there is a good correlation between the flow stress and the inverse of the length of the dislocation rosette size. Thus, once a rosette size versus flow stress calibration has been made for a particular crystal system, the

rosette size becomes a useful, simple, nearly nondestructive technique for determining the flow stress of a crystal.

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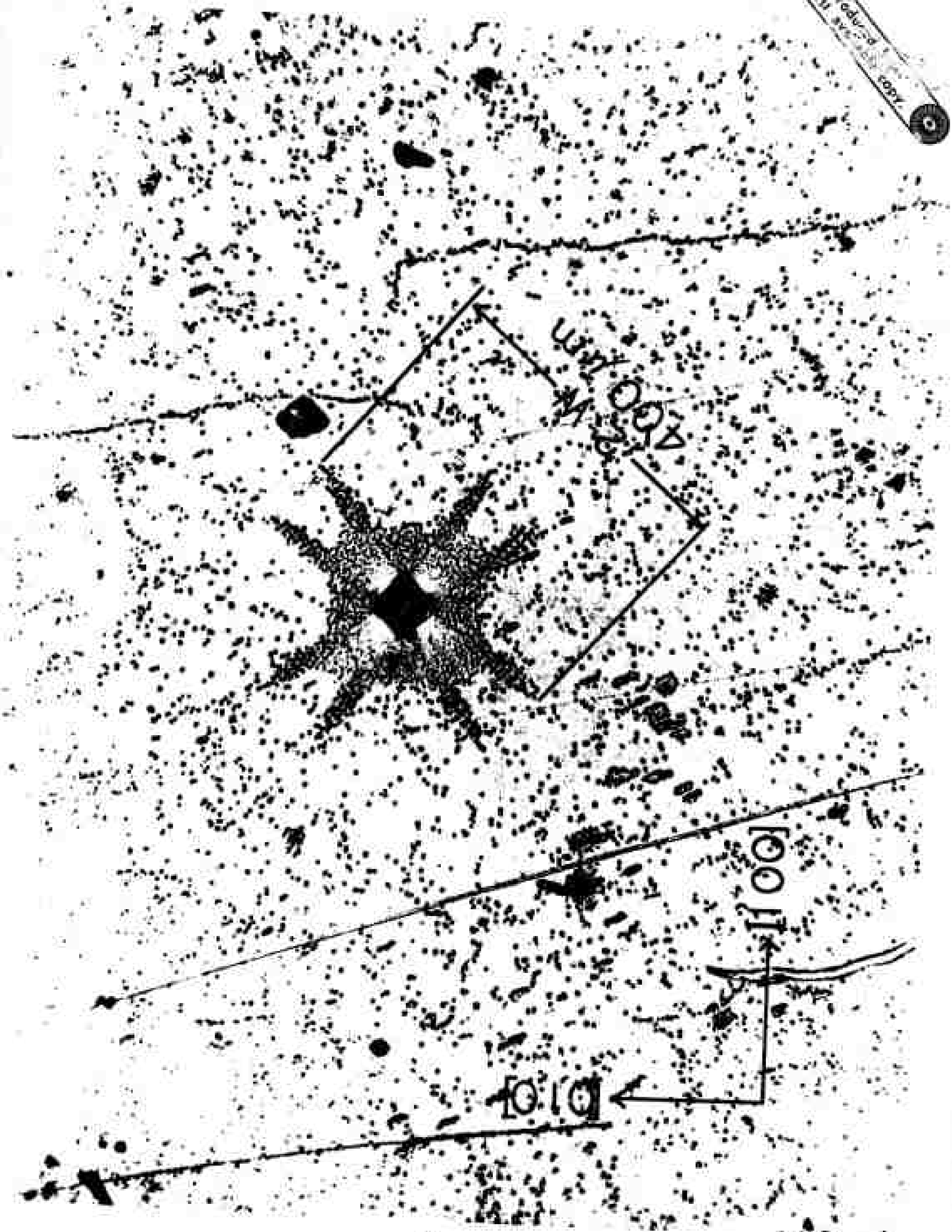
FIGURE CAPTIONS

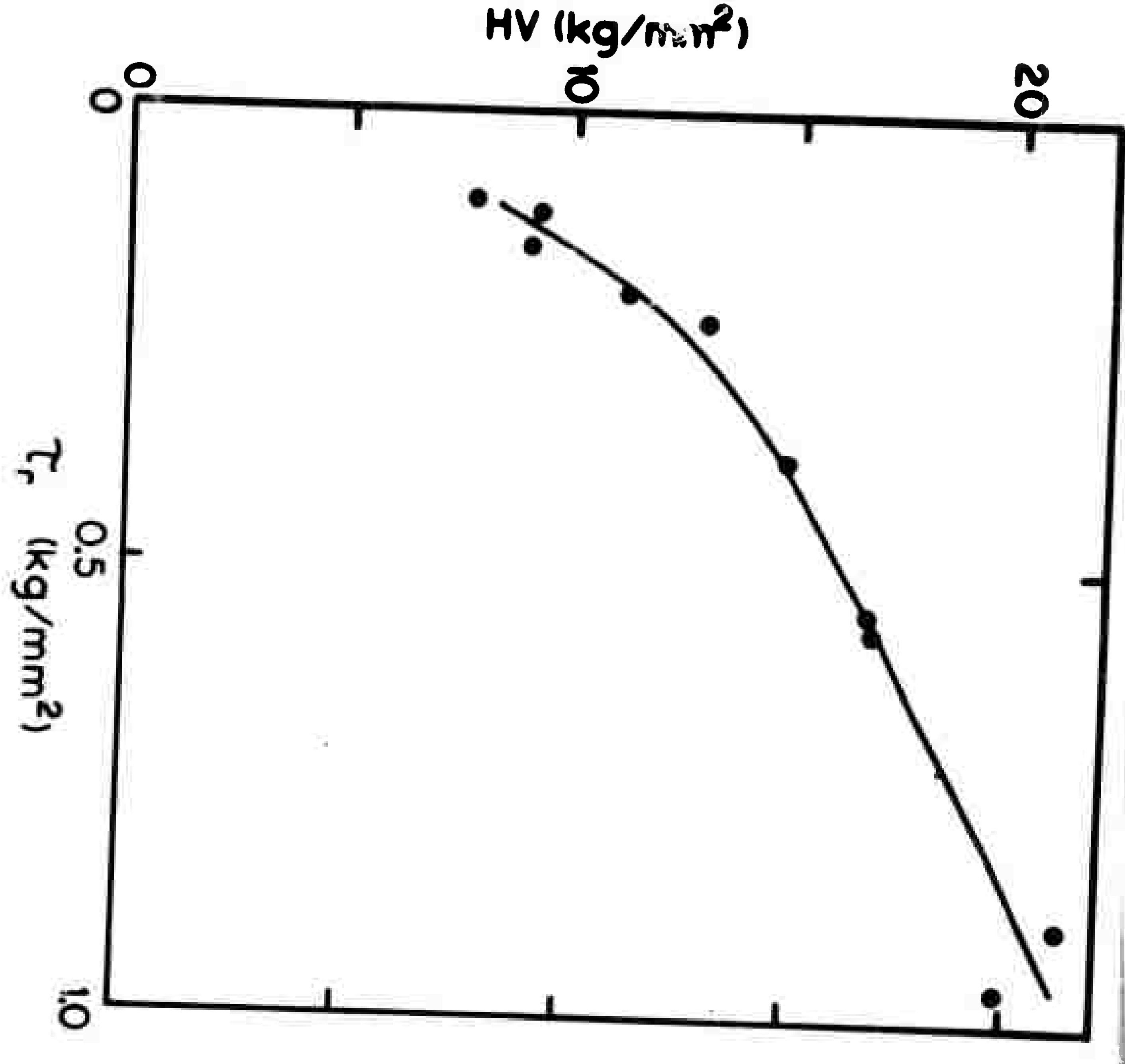
Fig. 1. A dislocation rosette of a 50 g weight indentation on KCl:Sr.

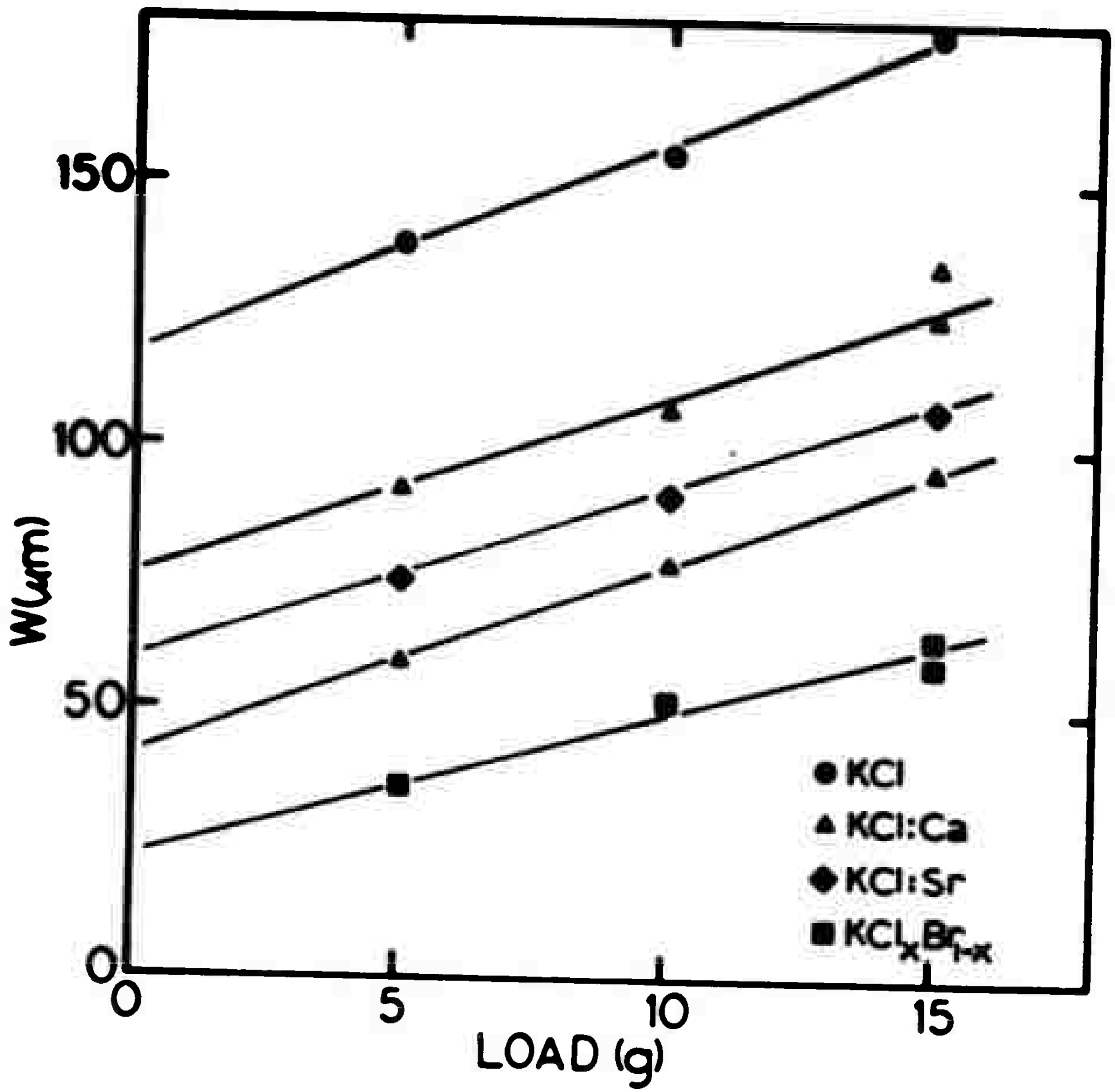
Fig. 2. Vickers hardness number versus resolved flow stress is shown.

Fig. 3. The dislocation rosette wing size, W, versus indenter load is shown.

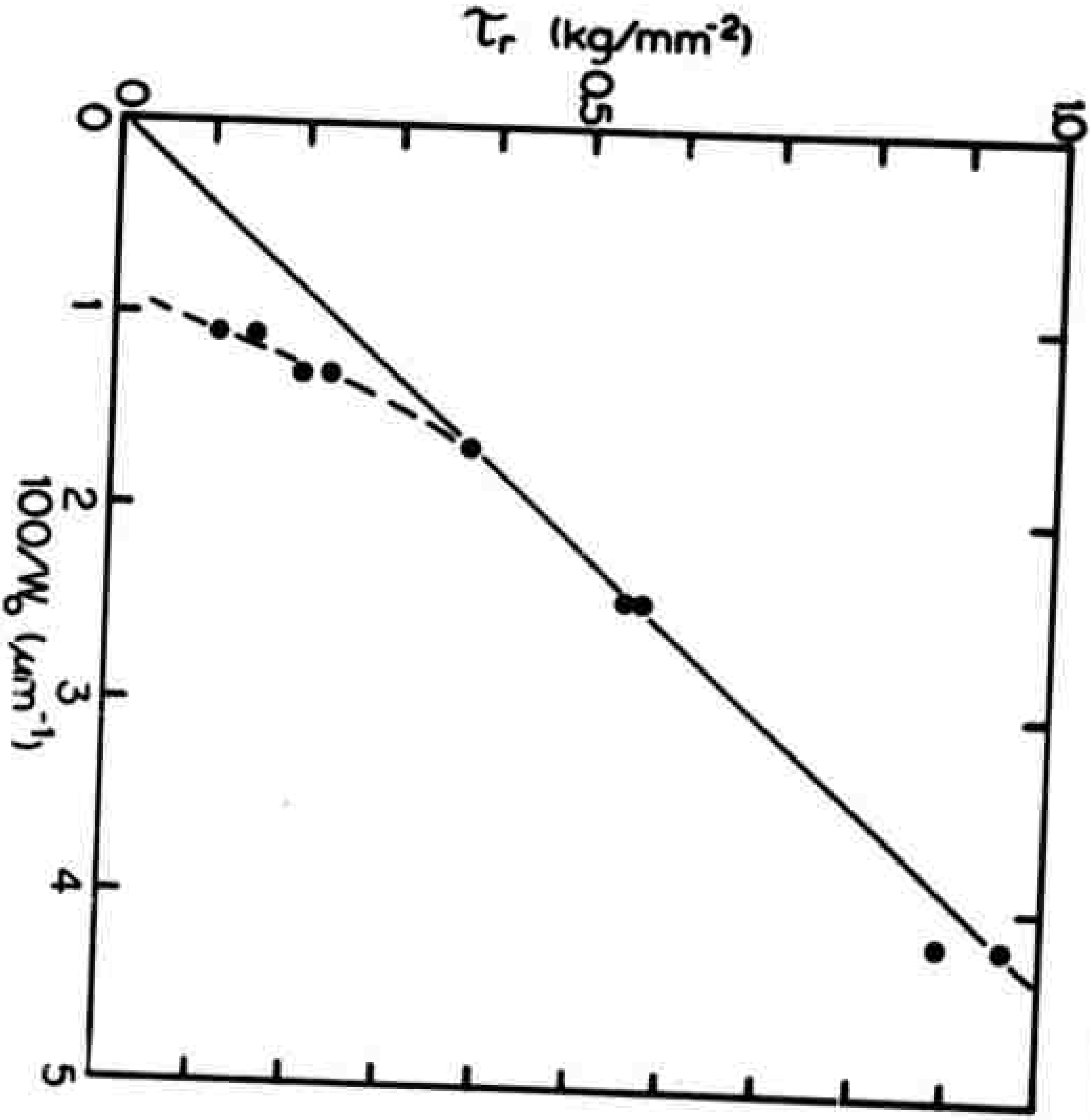
Fig. 4. This graph of resolved flow stress, τ_r, versus $100/W_o$ shows a linear relationship for the harder crystals.


400 μm
[001]
[010]


HV (kg/mm²)
0
10
20
τr (kg/mm²)
0
0.5
1.0



W(μm)
150
100
50
0
0
5
10
15
LOAD (g)
KCl
KCl:Ca
KCl:Sr
KCl$_x$Br$_{1-x}$



τ_r (kg/mm^{-2})
0
0.5
1.0
1
2
3
4
5
100/N_b (μm^{-1})